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**(54) Liquid crystal devices**

(57) A process for producing a rubbed polyimide alignment layer in a liquid crystal device, in which a polyimide alignment layer is deposited on a surface of the device, such as glass or germanium, the device is rubbed and subsequently heated, includes the steps of heating the polyimide layer so as to vary the liquid crystal tilt angle produced by the layer.

By varying the heat treatment, the optimum angle for a particular device may be selected.

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## SPECIFICATION

## Liquid crystal devices

5 This invention relates to liquid crystal devices.

In liquid crystal devices it is often required that at least one of the major surfaces of the body confining the liquid crystal materials, for example at least one of the opposing surfaces of a pair of parallel glass plates, be treated in such a way that the axes of the molecules of the liquid crystal material are not exactly perpendicular to, or not exactly parallel to the major surface, but the axes tilt uniformly with respect to the major surface at an angle known as the tilt angle. This tilt angle is usually achieved by applying a so called alignment layer to the major surface.

These alignment layers, in recent years, have taken the form of thin, typically in the region of 500 to 5000Å, layers of polyimide. These are applied to at least one of the major surfaces which are to confine the liquid crystal in the form of a solution, the resultant layer then being dried, cured and subsequently rubbed, with for example a linen cloth, in a chosen direction along the surface to achieve the necessary surface profile of the layer to align the liquid crystal molecules at a tilt angle.

A typical processing procedure for such a polyimide alignment layer would be to spin a solution of polyimide on the required surface, this being followed by drying the deposited solution for 30 minutes at 125°C. The thus deposited layer of polyimide would then typically be cured at 300°C, for two hours, this being followed by the rubbing process.

Such a procedure will typically produce a 1000Å thick layer of the cured polyimide. Tilt angles obtained, for example with the liquid crystal E7 produced by BDH in a liquid crystal device incorporating such a layer will typically be in the range 2.5° to 4.5° dependent on the exact polyimide used. It has not hitherto however been possible to vary the tilt angle produced by a particular polyimide.

Furthermore, recently a number of liquid crystal devices have been developed which require much larger tilt angles typically of 30° or more. Such large tilt angles have not hitherto been obtainable with rubbed polyimide layers. To obtain such large tilt angles it has been necessary to form alignment layers by depositing silicon monoxide by evaporation on the required surfaces at an angle of 5° to the surfaces. This latter procedure has the disadvantages of lower efficiency, higher cost, and lower reliability compared to rubbed polyimide alignment layers.

It is an object of the present invention to provide a process for producing a rubbed polyimide alignment layer in a liquid crystal device wherein the liquid crystal tilt angle produced by the layer may be varied, and

wherein it is possible to produce larger tilt angles than has been possible previously with rubbed polyimide layers.

According to the present invention a process for producing a rubbed polyimide alignment layer in a liquid crystal device comprising: depositing a rubbed polyimide alignment layer on a surface of the device; curing the layer; and rubbing the layer, is characterised in that the layer is heated so as to vary the liquid crystal tilt angle produced by the layer.

In one particular process in accordance with the invention, after depositing the layer, the layer is cured to an extent whereby the tilt angle is increased.

In another particular process in accordance with the invention, the layer is heated after it has been rubbed so as to decrease the tilt angle.

In such other particular process, all the curing of the layer may take place after rubbing of the layer.

Two processes for producing a rubbed polyimide layer in a liquid crystal device in accordance with the invention will now be described by way of example only.

In the first process to be described, a 3% solids solution of polyimide known by the Trade Name "Nolimid 32" and produced by Rhone Poulenc is spun onto a glass substrate which is to form a major confining surface for the liquid crystal material in the device, at 3000rpm for 30 seconds. The film thus formed on the substrate is subsequently dried at 125°C for 30 minutes to produce a 1000Å layer of polyimide on the substrate. The process so far is thus as in the previously known processes. The process in accordance with the invention subsequently differs from known processes however, in that the polyimide layer on the substrate is then cured for 12 hours at 300°C. After this extended curing process, the layer is then rubbed in the usual manner.

It is found that using the procedure for measuring tilt angles outlined in the article in Physics Letters, Volume 56A, number 2, pages 142-144, published March 1976 by G. Baur and V. Wittwer, the tilt angle for the BDH liquid crystal E7 produced by the polyimide layer is 11°. This is in contrast with the corresponding angle of 3.5° measured for a corresponding Nolimid 32 rubbed layer produced with a conventional curing process of 2 hours at 300°.

The inventors have hypothesised that the increased tilt angle is due to an increased hardness of the polyimide film due to the extended cure time. This is, however, a surprising result as the literature indicates that full imidisation of a polyimide layer is achieved within 2 hours at 300°.

It is found that the use of such a high tilt angle improves the performances of a super-twist dye display of the type described in

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Mol. Cryst. Liq. Cryst., Volume 123, pages 303-319 published in 1985 by Waters et al.

It will be appreciated that the use of higher curing temperatures than have previously been used will also produce increased tilt angles.

In the second process to be described, a 3% solids solution of Nolimid 32 is again spun onto a glass substrate at 3000rpm for 30 seconds, the film thus formed on the substrate being subsequently dried at 125°C for 30 minutes to produce a 1000Å layer of polyimide on the substrate. The polyimide layer is then rubbed, this being followed by a curing process at 300°C for 2 hours. It is found that the tilt angle for the liquid crystal E7 produced by BDH is zero. Good directional alignment of the liquid crystal is however produced.

In an adaptation of the second process, after the drying stage the polyimide layer is cured at 300°C for 2 hours before rubbing, the rubbed polyimide film then being cured for a further two hours at 300°C. It is then found that a tilt angle of 0.5° results, good directional alignment of the liquid crystal again being produced.

In a further adaptation of the second process, after the drying stage the polyimide layer is cured at 300°C for 2 hours before rubbing, the rubbed polyimide film then being heated for a further two hours at 200°C.

This process produces a tilt angle of 2.5° with good directional alignment of the liquid crystal.

Thus by variation of the heat treatment of the polyimide layer as described herebefore the optimum tilt angle for a particular liquid device crystal device may be selected.

It will be appreciated that whilst the polyimide layer described in the examples in accordance with the invention is Nolimid 32, many other polyimide materials may be used in a process in accordance with the invention. These include the Electro Science Laboratories materials 55 HS and 55 HD and the Merck material ZL12650.

It will also be appreciated that a process in accordance with the invention may be used in the manufacture of liquid crystal devices incorporating a selection amongst many different liquid crystal materials.

It will also be appreciated that whilst in the processes described herebefore by way of example, the polyimide layer is deposited on a glass substrate, many other materials may be used to confine the liquid crystal material, for example germanium.

#### CLAIMS

1. A process for producing a rubbed polyimide alignment layer in a liquid crystal device comprising: depositing a rubbed polyimide alignment layer on a surface of the device; curing the layer; and rubbing the layer, characterised in that the layer is heated so as to vary the liquid crystal tilt angle produced by

the layer.

2. A process according to Claim 1 in which after depositing the layer, the layer is cured to an extent whereby the tilt angle is increased.

3. A process according to Claim 1 in which the layer is heated after it has been rubbed so as to decrease the tilt angle.

4. A process according to Claim 3 in which all the curing of the layer takes place after rubbing of the layer.

5. A process for producing a rubbed polyimide alignment layer in a liquid crystal device substantially as hereinbefore described.

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